

In the Specification:

Please delete the title and insert the following title:

Precise Flow-Oriented Multi-Angle Remission Sensor.

Page 1, before the first paragraph, insert—Cross Reference to Related Applications

This application is a National Phase Application of PCT/EP2004/014603, filed 22. December 2004, which claims priority to DE103 61 058.8, filed 22. December 2003.

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effect can be produced by means of metallic pigments, for example platelet-like aluminum flakes. Interference effects can be achieved by means of so-called interference pigments. These are normally platelet-like particles of a virtually transparent substrate material, for example mica, with a refractive index of the order of magnitude of the surrounding binder matrix, the outer surfaces being finished with an optically very highly refractive coating, for example of metal oxides. If metallic pigments and/or effect pigments are added to a coating (generally in addition to their colorants), then (desired) effects with considerable anisotropy are produced for an observer. This is because the lightness and chromaticity varies as a function of the viewing direction (goniochromatic effect). In the case of the effect pigments, a variation in hue also occurs. The optical properties, in particular the reflectance of liquid samples of such effect coatings, that is to say coatings which contain non-isometric particles, and other liquid samples which contain non-isometric particles, thus depend on the orientation of these non-isometric particles in the liquid sample.

For correct, reproducible measurements, in particular reflectance measurements of liquid samples which contain non-isometric particles, alignment of the particles before the measurements is thus necessary. In the case of needle-like particles, alignment in one axis is in principle sufficient. For the correct measurement of samples which contain platelet-like particles, for example metallic pigments and/or effect pigments, alignment in two axes is required. In the analysis systems known in the prior art for liquid samples containing particles, such alignment of the samples is not carried out.

It is therefore an object of the present application to provide an analysis system for the measurement, in particular reflectance measurement, of liquid samples which contain non-isometric particles, and to provide an apparatus for aligning non-isometric particles in a liquid sample, in particular in a liquid pigment preparation.

This object is achieved by a three-dimensional flow cell for aligning non-isometric particles in a liquid sample in two axes, comprising a feed zone for the sample containing particles to be aligned and an outlet for the sample containing particles aligned in two axes, a fluid element of the sample with the dimensions  $a$ ,  $b$ ,  $c$  being transformed in an expansion zone into a fluid element with the dimensions  $a \times n$ ,  $b/(n \times m)$ ,  $c \times m$ ,  $a$  being the width,  $b$  the height and  $c$  the length of the fluid element and  $n$  and  $m$  being constants (degree of expansion) which depend on the geometry of the flow cell and which signify positive numbers  $\geq 1$ .

The liquid sample containing non-isometric particles concerns dispersions. Preferred liquid samples which contain non-isometric particles are liquid pigment preparations. Such liquid

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distribution with a distributor comprising tubes or hoses (for example a Y piece or T piece from 1 to 2).

In the simplest form, the flow volume is symmetrical with respect to a mid-plane. In this case, however, use must be made of a window which is rectangular and no longer than the measuring zone. In the preferred use of a large planar plate and a measuring cell that can easily be removed, the flow volume has to be modified, in that the inlet and outlet volumes are bent away from the plane of the plate relative to the measuring zone. One possible design in particular bends so far that the planar surface on the sensor side of the inlet/outlet volume coincides with the planar plate. A bend which goes even further is more advantageous, so that there is still a wedge of cell material between planar plate and flow volume (a wedge angle of  $5^\circ$  to  $30^\circ$  is advantageous,  $15^\circ$  to  $25^\circ$  is particularly advantageous), and only the measuring zone is bounded directly by the plate. As a result, only a small part of the plate is touched by the product.

If a fluid element of dimensions  $a, b, c$  is deformed ( $a$  width,  $b$  height,  $c$  length), because a flow cross section  $A, C$  is transformed into  $n \times A, C \times m$ , the result is a fluid element  $a \times n, b/(n \times m), c \times m$ . The angles or their tangent in the  $a, b$  plane are varied by  $1/(n \times n \times m)$ , the angles in the  $c, b$  plane by  $m^2/n$ . An equivalent alignment in both axes is preferred, that is to say preferably  $(n \times n \times m) = (m \times n \times m)$ , respectively  $n = m$ , and then both factors are  $n^3$ . Thus, for example with  $n = 5$ , an entry cross section of  $A=4, B=25$  is transformed into an outlet cross section of  $A=20, B=1$  and aligned in both axes by the factor 125.

The achieved, defined alignment of the non-isometric particles, and also the defined deformation of the fluid element (alignment of macromolecules) can be used with various optical and non-optical measuring methods for determining further sample properties. In addition to the reflectance measurement known from colorimetry, other photometric arrangements (for example transmission, laser diffraction) and imaging optical methods (for example image analysis, backscatter probes) can be employed.

$n$  and  $m$  are the respective level of expansion of the fluid element. The absolute values for  $n$  and  $m$  depend, inter alia, on how severe the deformation of the fluid elements of a flow is intended to be. The severity of the deformation is in this case dependent on the intended application and on the size of the non-isometric particles in the liquid sample. In general,  $n$  is 1.5 to 7, preferably 2 to 5, particularly preferably 3 to 5, quite particularly preferably 4 to 5, the preferred values being suitable in particular when the flow cell according to the invention is used in photometric measuring devices, in particular reflectance sensors. When

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the flow cell according to the invention is used in image analysis, for example, other values for  $n$  can be preferred.  $m$  is preferably  $n$ , as already explained above.

The thermal motion, turbulence and rotational forces in the event of shear gradients counteract this alignment. Turbulence can be avoided by a suitable flow velocity to be determined without difficulty by those skilled in the art with a given geometry of the three-dimensional flow cell according to the invention. The rotational forces have a weaker effect the flatter the alignment is.

The deformation according to the invention is thus preferably carried out on a path which is sufficiently short to minimize the formation of a flow profile, but the decay of the alignment as a result of the thermal motion is at the same time sufficiently long for no sharp deflections of the flow to take place. In the adjacent parallel part (the measuring zone) as well the path length is chosen to be no longer than necessary, in order to minimize thermal diffusion and the formation of a flow profile. The parallel part merely has to be sufficiently long to accommodate completely the "measurement surfaces" which result from the beam cross section and angle of incidence. The measuring zone is preferably 2 to 10 mm, particularly preferably 4 to 8 mm, long. One advantageous variation is a second measurement at a greater distance, for example 10-20 mm, in order to register the extent of the decrease in alignment as a product property. Instead of varying the distance, a defined variation in the flow velocity can also be employed for this purpose.

The level of alignment itself depends to a first approximation on the expansion ratio, that is to say the flow velocity is selected such that still no turbulence occurs but is as high as possible, in order that the thermal disordering is minimized and the shear forces in the measuring gap (measuring zone) keep the surfaces that touch the product clean. With a given length of the measuring gap (measuring zone), a suitable flow velocity above the pressure loss is set (0.1 to 3 bar, preferably 0.5 to 1.5 bar). The volume flow is then measured, the flow velocity is calculated and checked for turbulence.

If the three-dimensional flow cell according to the invention is part of a photometric measuring device, then the liquid sample aligned in two axes strikes the measuring window (measuring zone) of the photometric measuring device directly at the end of the expansion zone.

A further subject is a method of aligning non-isometric particles in a liquid sample, the liquid sample flowing through a three-dimensional flow cell according to the present

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application, a fluid element of the liquid sample with the dimensions  $a$ ,  $b$ ,  $c$  being transformed into a fluid element with the dimensions  $a \times n$ ,  $b/(n \times m)$ ,  $c \times m$ ,  $a$  signifying the width,  $b$  the height and  $c$  the length of the fluid element and  $m$  and  $n$  being constants which depend on the geometry of the flow cell and which signify positive numbers  $\geq 1$ .

In the method according to the invention, it is preferable if  $1/(n \times n \times m) = 1/(m \times n \times m)$ . Preferred values for  $n$  have already been mentioned above.

Suitable liquid samples with non-isometric particles, and suitable non-isometric particles and suitable flow velocities at which the liquid sample flows through the three-dimensional flow cell have likewise already been mentioned above.

A further subject of the present application is the use of the three-dimensional flow cell according to the invention for the two-dimensional alignment of non-isometric particles in a liquid sample, preferably for the alignment of non-isometric particles in liquid pigment preparations.

The form of the flow cell, comprising a cross section-deforming feed zone, an expansion zone, a measuring zone running in parallel and an outlet has been described above. The mechanical construction of such a flow cell depends on the requirements specific to its use. For the preferred reflectance measurement, a particularly advantageous construction is implemented by a planar plate and a three-dimensional molding, which has a feed opening, a measurement opening for the fitting of the measuring window and an outlet opening. A suitable material is preferably metal or plastic, particularly preferably stainless steel and Teflon. This fabrication combines reproducible precision and easy cleaning.

The production of the flow cell is carried out in accordance with methods known to those skilled in the art, for example by boring, grinding or milling the flow path into a block of one of the aforementioned materials. Furthermore, the flow cell can be produced by injection molding if the material of the flow cell is suitable for injection molding.

A further advantageous fabrication technique is the press forming of plastics, preferably Teflon. This means that, by using a shaped plunger (tool), a basic volume can be pressed into a block and the necessary flow guidance can be achieved by simple, inserted displacement elements. The displacement elements are convex and can therefore be fabricated without difficulty using conventional methods.

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run parallel to the measuring window. Here, a fluid element belonging to the liquid sample and having the dimensions  $a$ ,  $b$ , and  $c$  is transformed into a fluid element having the dimensions  $a \times n$ ,  $b/(n \times m)$  and  $c \times m$ ,  $a$  being the width,  $b$  the height and  $c$  the length of the fluid element and  $n$  and  $m$  being constants which depend on the geometry of the flow cell and which signify positive numbers  $\geq 1$ . Preferred embodiments of the three-dimensional flow cell and values for  $n$  and  $m$  have already been mentioned above. When selecting the cross sections and expansion coefficients ( $a$ ,  $b$ ,  $c$ ,  $n$ ,  $m$ ), a suitable shearing gap (= measuring zone) must be set.

Figure 7 illustrates a preferred embodiment of a reflectance sensor having a sample analysis unit (B) for the reflectance measurement of liquid samples containing non-isometric particles, comprising the measuring window (Ba) and the sample analysis cell with three-dimensional flow cell (Bb) and also a holder for the fiber optics (Ab) of the optical unit (A).

*Figure 7: Reflectance sensor with three-dimensional flow cell for measuring liquid samples containing non-isometric particles*

Here:

- 1 is the baseplate (mounting plate)
- 2 is the holder for the measuring window
- 3 is the measuring window
- 4 is the opening for the fiber system
- 5 is the drip edge
- 6 is the basic product cell body
- 7 is the product outlet
- 8 is the product feed with specific three-dimensional form for the alignment
- 9 is the shearing gap

The sample analysis cell can be sealed off with respect to the optical unit in accordance with all the methods known to those skilled in the art. The considerable shearing of the product in the shearing gap is an important factor both in order to obtain a defined sample state, that is to say by means of this shearing agglomerates of pigment particles, for example, are broken up, and also to achieve self-cleaning of the measuring window, which is continually freed of particles possibly remaining caught on the measuring window by the intense shearing of the sample.